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A.U.W.E. Tech Note 192/65

AUGUST 1965

Part 3

A CORROSION STUDY ON MATERIALS
FOR USE WITH PHOSPHORUS & SULPHUR
BEARING FUELS

Part 3

[C]

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BY
H. GOLDIE

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A CORROSION STUDY ON MATERIALS
FOR USE WITH PHOSPHORUS AND SULPHUR BEARING FUELS.

PART THREE

EXAMINATION OF COMBUSTION CHAMBER COMPONENTS ETC.

(9) Technical note, (10) H. Goldie.

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N.D. Committee for Prevention of Corrosion and Fouling; Machinery Sub-Committee	57 to 86
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A CORROSION STUDY ON MATERIALS
FOR USE WITH PHOSPHORUS AND SULPHUR BURNING FUELS

PART THREE

EXAMINATION OF COMBUSTION CHAMBER COMPONENTS

PRECIS

1. The protection offered by oxide films and corrosion product during the static laboratory tests was not reproducible under combustion conditions. The action of fast-moving gas and/or liquid droplets of metaphosphoric acid in the combustion system led to extremely high metal loss by erosion as well as corrosion.
2. This part of the report includes a microscopical study of sections taken from damaged components after exposure to phosphorus-phosphorus sesquisulphide (PPS) combustion products. The examinations were carried out at various stages of the PPS programme, principally to give some indication as to the cause of damage.
3. The report shows that damage can be caused by
 - (a) corrosion and erosion of materials by liquid phosphoric acid contained **in the moving combustion stream**. Products of the reaction were found to be metallic metaphosphates and hydrogen.
 - (b) corrosion and erosion of materials by phosphoric acid vapour, the products of reaction being phosphides or phosphide eutectics depending on the distribution of phosphorus.

CONCLUSIONS

4. The mechanisms of attack associated with metals when exposed to the phosphorus-phosphorus sesquisulphide (PPS) combustion gases were found to be similar to those shown by metals when exposed to the synthetic combustion atmospheres used for the static laboratory tests.
5. The protection offered by adhering corrosion product during static tests was not reproducible under combustion conditions. The action of fast-moving gas and/or liquid droplets of metaphosphoric acid in the combustion chamber led to high loss of metal by erosion as well as corrosion.
6. Metaphosphoric acid vapour was considered responsible for the production of metallic phosphides. Combustion trials lasted only a matter of minutes. Even so, the metallic scales were found to contain sufficient phosphorus to support phosphide formation.
7. The short periods of exposure led to uneven distribution of phosphorus in the attacked areas and eutectic distribution was not so characteristic as compared with the results from the much longer exposures associated with laboratory tests.

RECOMMENDATIONS FOR FUTURE WORK

8. Future work should include the use of

- (a) micro-analysis techniques to identify surface and grain boundary reactions of alloys when exposed to the PPS combustion products,
- (b) X-ray diffraction techniques to identify deposits and corrosion products formed.

Future work should also include

- (c) combustion trials using test samples of metals which form solid scales or corrosion product up to 1500°C e.g. Mo, Ta, Zr, Ti.
- (d) Phosphorus-phosphorus sesquisulphide combustion trials using test samples of conventional materials coated with non metallic deposits e.g. alumina, silicon nitride and also with the above resistant metals and their alloys using vapour deposition or electro-deposition as appropriate.

INTRODUCTION

9. In addition to corrosive atmosphere and working temperature, factors such as flow rate of working fluid, non-equilibrium associated with practical combustion conditions and pressure within the system, must be included in the overall assessment of material or component damage.

10. The protection offered by adhering corrosion product during the static laboratory tests was not reproducible under practical combustion conditions. The corrosion product when formed was readily removed by the action of the gas stream. This affect led to conjoint attack, i.e. erosion and corrosion.

11. The report is based on a microscopical study of the surface and structural characteristics of combustion chamber components after high-temperature exposure to the phosphorus-phosphorus sesquisulphide (PPS) combustion products. The examinations were carried out at various stages of the PPS programme principally to give some indication as to the case of damage.

EXAMINATION 1: HIGH TENSILE STEEL SCREW, CADMIUM PLATED

Examination

1. The head and last seven threads had been affected, the appearance being that of an eroded surface (Figure 1). The circumference of the head had been reduced and the socket enlarged to such an extent that the wall corresponding to one side of the hexagon had been removed almost completely (Figure 2).

13. Microscopical examination showed the material to have a uniform martensitic type structure. A hardness survey along the length of the screw gave DFN 460 maximum, DFN 440 minimum indicating the absence of over-heating or any appreciable temperature gradient during the trial. A thin scale covered the head and damage threads of the screw. There was no evidence of intercrystalline attack.

Conclusion

14. It is considered that the set screw had suffered damage by corrosion/erosion. Erosion by flowing acid probably contributed the major part of the damage.

EXAMINATION 2: STAINLESS STEEL THERMOCOUPLE

Examination

15. Figure 3 shows that an area of the probe approximately 0.7 inch long and half the circumference wide had a highly-polished appearance. Approximately at the centre line of this area and adjacent to the tip of the probe a longitudinal hole had been formed.

16. Further examination revealed that the wall thickness had been considerably reduced at the polished area, the area of maximum reduction resulting in a hole. The down stream side of the probe appeared heavily pitted, probably due to a form of abrasive blasting by extremely fine combustion product particles.

17. The thermocouple probes were not sectioned for microscopical examination.

Conclusion

18. It is considered that failure of the thermocouple probe was principally due to erosion caused by fine combustion product in the gas stream. The flow of liquid droplets of phosphoric acid would also produce a polished and rounded surface.

EXAMINATION 3: STAINLESS STEEL DILUENT RING (EN58J) TEST RUN NO. 28

Test Conditions

Duration of hot run	-	130 seconds
Mean final gas temperature	-	700°C
Fuel/Oxidant Ratio	-	5 per cent fuel rich.

Examination

19. The mouth and bore of the diluent ring were covered with a green deposit of inorganic phosphate. The rear face was partially covered with a dark adherent metallic scale. The scale was subsequently found to be magnetic. The conical approach wall and areas adjacent to the water injection holes had been badly damaged (Figure 4). The rear face also suffered damage (Figure 5), the corrosion/erosion pattern being dependent on the location of the water injection holes.

20. Microscopical examination of sections cut from

(a) conical wall approach

(b) areas adjacent to the injection holes and rear face

showed evidence of a metallic scale containing entrapped particles of phosphate (Figure 6). Areas within the scale showed dendritic characteristics associated with the freezing of liquid metal (Figure 7). Subsequent etching revealed the phosphorus rich character of the metallic scale but a definite eutectic phase could not be clearly resolved (Figure 8).

21. These samples were examined using the Electron Beam X-Ray micro-analyser. The results are detailed below. X-ray images showing the distribution of Fe, Ni, Cr, Cu, S and P at the scale/base metal interface are shown in Figures 9 to 13 respectively.

22. The examination gave the following information. A slow scan for nickel across a line in the sample indicated that the nickel concentration increased in the plain areas as compared to those areas in which a eutectic type structure appeared. The concentration of iron in these plain areas was also found to be high while that of phosphorus appeared to be lower.

23. The concentration of chromium in the phosphorus-rich region or scale was low and no marked concentration gradients were found in the stainless steel at the base metal/scale interface.

24. Appreciable quantities of copper were found in the scale generally evenly distributed. Small quantities of sulphur were detected mainly segregated at the metal/scale interface. The approximate concentrations of Phosphorus, Copper and chromium in the scale were estimated to be 10, 4, and 3, respectively. The attack on monel metal components up stream of the diluent ring can explain the presence of liquid copper phosphide eutectic entrapped in the combustion stream and subsequent presence of this metal in the attacked surfaces of an alloy component originally free from copper.

Conclusions

25. It is considered that the damage to this component was caused principally by high-temperature corrosion from phosphorus compounds.

26. The microscopical examination revealed the presence of surface phosphides and phosphide eutectics. Micro-analysis confirmed the presence of phosphorus in sufficient quantity to support eutectic phosphide formation.

27. These findings suggest that during the extremely short exposure (130 seconds) the surface of the diluent ring exceeded 950°C, i.e. the minimum temperature for eutectic formation. Liquid acid attack may have contributed to the total damage suffered by the component but this effect would only be of minor importance compared with phosphide formation.

EXAMINATION NO. 4: TEST RUN NO. 32

<u>Component</u>	- Supersonic Nozzle.
<u>Material</u>	- IN58J Stainless Steel protected with 0.003 inch gold.
<u>Test Conditions</u>	- Duration of trial, 120 seconds. Mean final product gas temperature, 400°C Fuel/Oxidant ratio : 5% fuel rich.

Examination

28. The upstream surface of the nozzle was covered with a green deposit of metaphosphate (Figure 14). The throat area showed evidence of coating failure due to blistering. The exhaust area was badly blistered and showed deposits of solid corrosion product. The rear face of the nozzle was also damaged (Figure 15).

29. Metallurgical examination revealed that the blistering may have resulted from the effect of heat on poor quality plating. Irregular pile up of gold 0.030 inch thick appeared on the downstream surfaces. Areas were visible where the gold had been completely removed leaving the base metal exposed to the combustion gases.

Conclusions

30. The examination failed to show positive evidence of direct chemical reaction between combustion phases and the protective gold. It is considered that the damage to this component was caused by

- (a) chemical attack of the base metal through cracks and pores in the protective coating,
- (b) the action of the fast moving gas/liquid acid stream over the soft gold layer.

31. However it is difficult to account for the large pile up of gold on the downstream surfaces unless the temperature within the chamber was higher than 400°C.

EXAMINATION NO. 5: TEST RUN NO. 39

Components

- (a) Experimental Blade: EN58J Stainless Steel.
- (b) Circular Rod used as 'Control': Silver Steel.

Test Conditions

- (a) Duration of trial: 123 seconds.
- (b) Mean final gas temperature: 1000°C.
- (c) Fuel/Oxidant Ratio: 5/3 Fuel Rich.
- (d) Components exposed to the jet efflux from the combustion chamber.

Stainless Steel Blade

32. The leading edge of the blade suffered severe erosion while the centre had been virtually removed leaving a vee shaped groove. The apex of the groove almost reached the opposite edge of the blade (Figure 16).

33. Microscopical examination showed an almost continuous non-adhering metallic scale at the damaged surfaces (Figure 17). Certain areas showed evidence of intercrystalline attack and deposits of metaphosphate associated with the metallic scale, (Figure 18). Subsequent etching revealed that the scale contained eutectic phosphides (Figure 19).

Conclusions

34. It is considered that the damage to this component was caused by reaction between base metal and phosphoric acid (probably metaphosphoric vapour) ultimately resulting in the formation of low melting point phosphides. These compounds once formed would be rapidly removed by the action of the gas stream.

Silver Steel used as "Control"

35. The tip of the exposed end had been completely removed while the remainder appeared to be covered with a dark metallic deposit. This deposit was subsequently found to be extremely hard and porous (Figure 20).

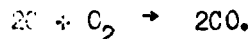
36. Microscopical examination of a longitudinal section cut from the rod revealed a rapid change in microstructure between unexposed and exposed areas. The former showed a spheroidised structure related to that of the original condition of the material. The latter was found to consist of coarse martensite, a structure normally associated with heating to temperatures above the upper critical followed by rapid cooling. The deposit was found to contain primary dendrites of martensite in a fine eutectic matrix. Figure 21 shows the structure after etching at the steel/deposit interface.

37. Electron Beam X-Ray microanalysis of the deposit showed that the iron content of the scale was 10% less than that of the base metal. The phosphorus and copper contents were found to be approximately 1%. Figure 22 shows the electron image of the area selected for analysis. Figures 23 and 24 are X-ray images for phosphorus and iron respectively.

Conclusions

38. Although phosphorus was found in the deposit, the eutectic matrix was considered to be essentially transformed Ledeburite. This structure probably resulted from a temperature exceeding the equilibrium liquidus i.e. for silver steel approximately 1350°C.

39. The porous nature of the deposit may have resulted from a gas-forming reaction similar to that of "carbon boil" in steel making. This would require removal and oxidation of carbon from the silver steel/deposit to form carbon monoxide by the reaction



40. The temperature at the top of the rod may also have been raised above the gas stream by combustion of iron to phosphide.

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HIGH TENSILE SCREW

FIGS 1 & 2

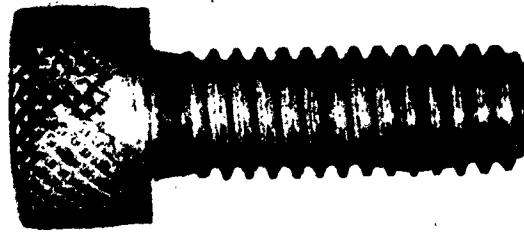
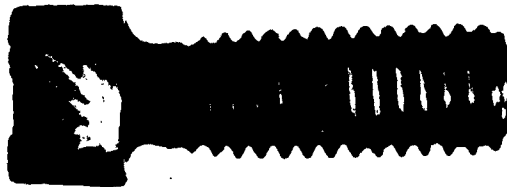


FIG 1



X 3.5

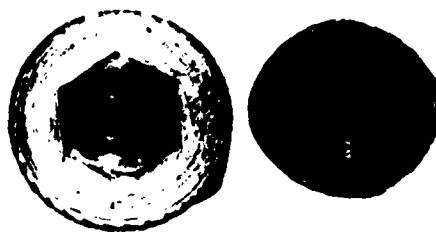
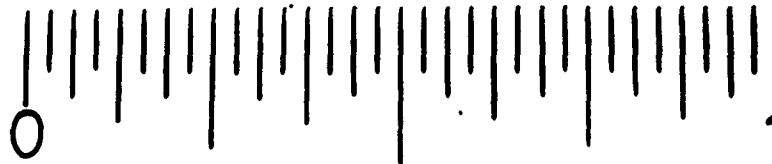


FIG 2



X 3.5

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FIG 3

STAINLESS STEEL THERMOCOUPLE

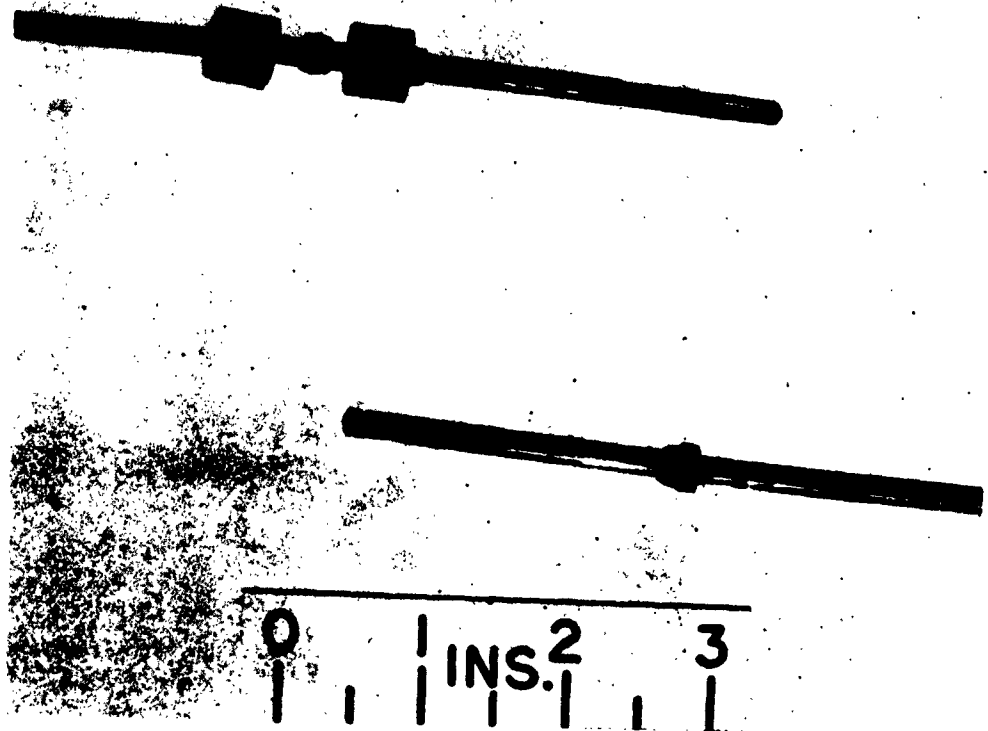


FIG 3

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FIG 4
UPSTREAM FACE

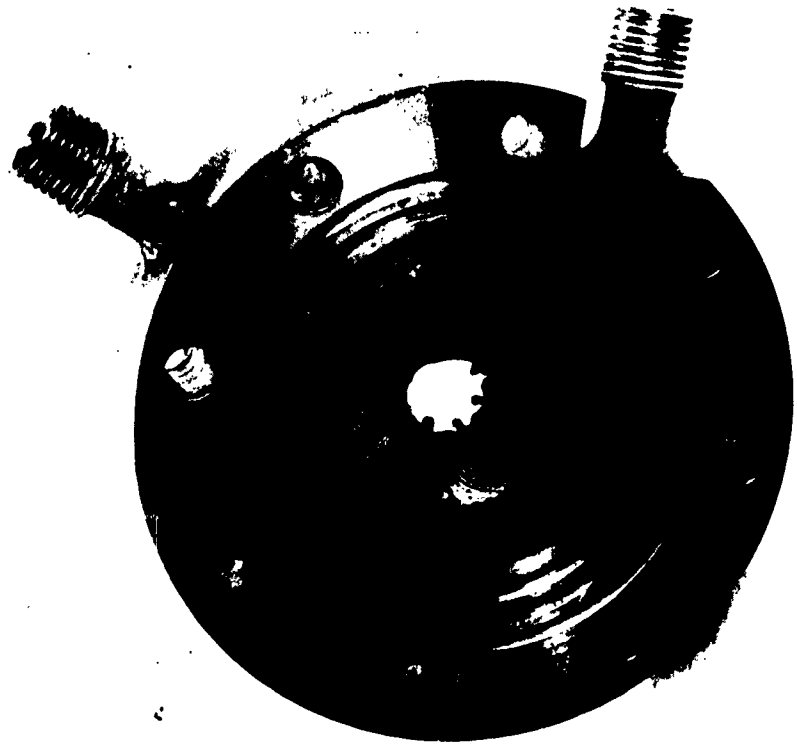
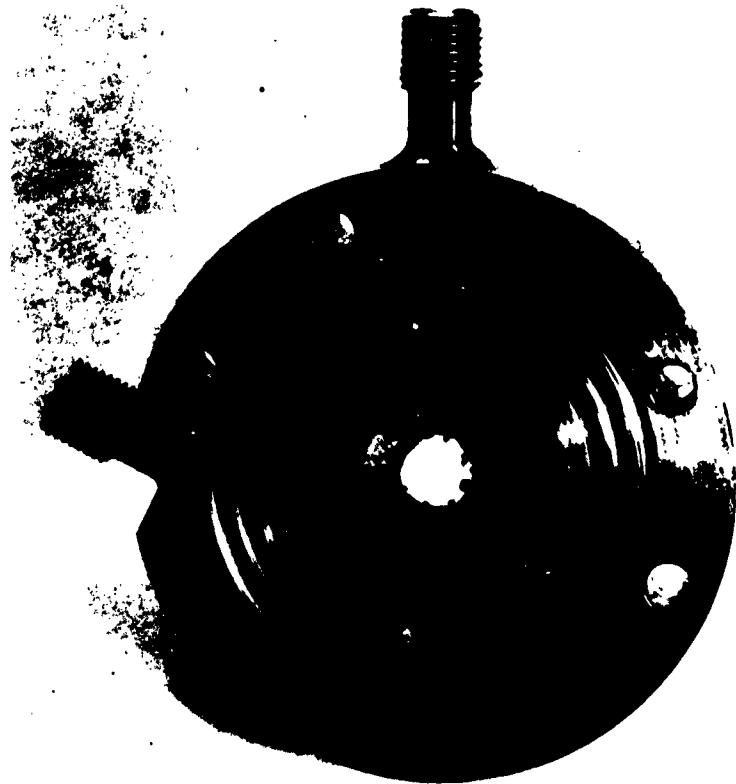


FIG 5
DOWNSTREAM FACE



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FIGS 6&7

TEST RUN No. 28
DILUENT RING

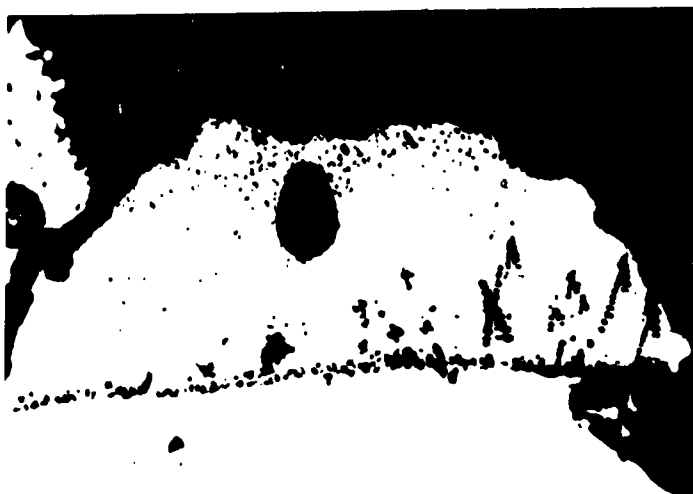
FIG 6



X400

UNETCHED

FIG 7



X400

UNETCHED

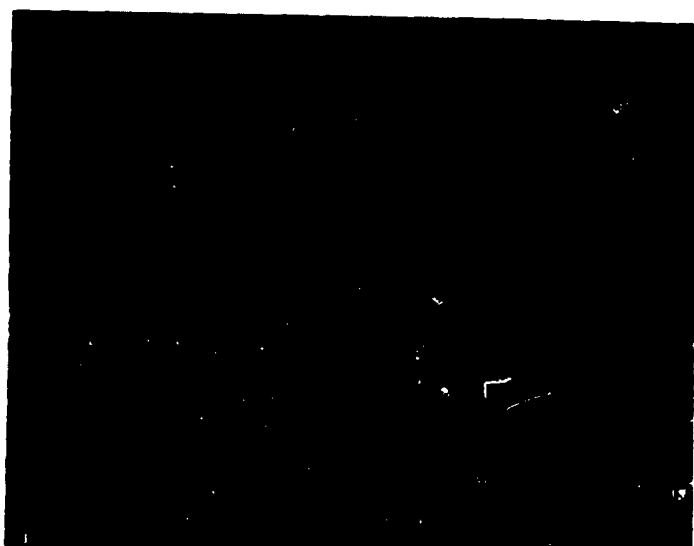
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FIG 8

TEST RUN-28
DILUENT RING

FIG 8



X400

ETCHED 10% OXALIC ACID ELECTROLYTIC

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TEST RUN - 28
STAINLESS STEEL DILUENT RING
MICRO ANALYSIS

FIG 9



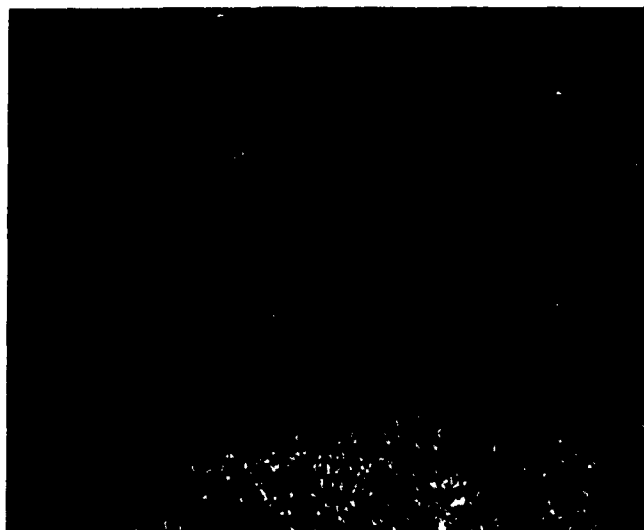
← SCALE

← BASE METAL

X960

X-RAY IMAGE - NICKEL

FIG 10



← SCALE

← BASE METAL

X960

X-RAY IMAGE CHROMIUM

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TEST RUN-28
STAINLESS STEEL DILUENT RING
MICRO ANALYSIS

FIG 11



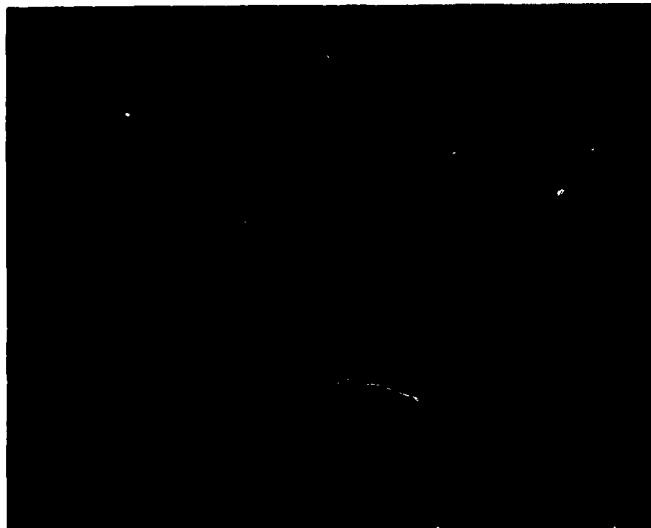
← SCALE

← BASE METAL

X960

X-RAY IMAGE COPPER

FIG 12



← SCALE

← BASE METAL

X960

X-RAY IMAGE SULPHUR

TEST RUN - 28
STAINLESS STEEL DILUENT RING
MICRO ANALYSIS

FIG 13



X 900

X-RAY IMAGE PHOSPHORUS

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TEST RUN-32
SUPERSONIC NOZZLE - GOLD PLATED

FIG 14



UPSTREAM FACE

FIG 15



DOWNSTREAM FACE

TEST RUN - 39

EXPERIMENTAL TURBINE BLADE EN58J
PLACED IN EFFLUX FROM NOZZLE

JET DIRECTION



FIG 16

x5

TEST RUN-39
EXP BLADE-EN58J

FIG 17



UNETCHED

X500

FIG 18



UNETCHED

X500

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TEST RUN-39
EXP BLADE-EN58J

FIG 19



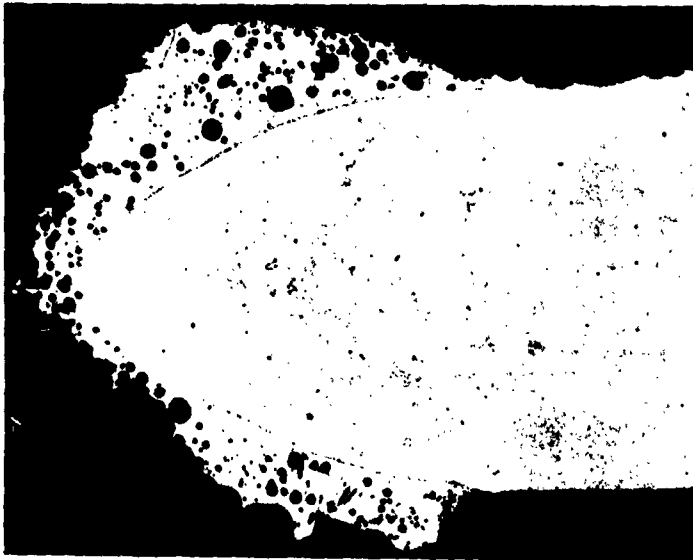
X 500

ETCHED-10% OXALIC ACID ELECTROLYTIC

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TEST RUN-39
CONTROL ROD - SILVER STEEL

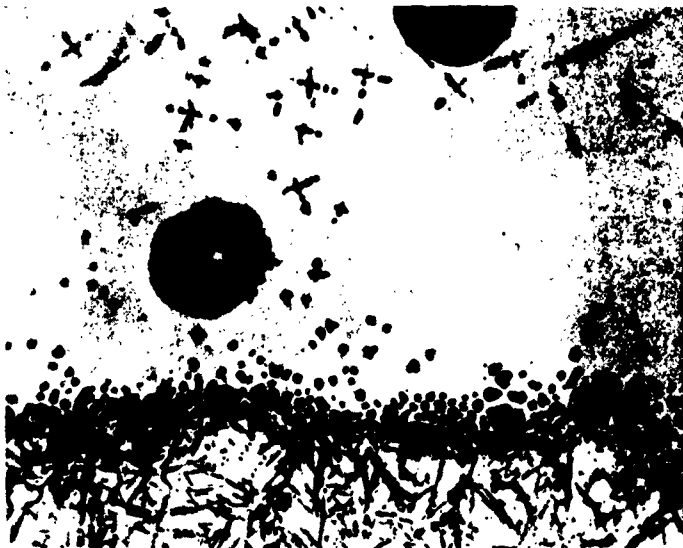
FIG 20



x5

UNETCHED

FIG 21



x250

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SILVER STEEL ROD

FIGS 22,23 &24

FIG 22

BASE METAL →



ELECTRON IMAGE
X 750

← SCALE

FIG 23



X-RAY IMAGE
X 750
IRON

FIG 24



X-RAY IMAGE
X750
PHOSPHORUS

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Examination of Combustion Chamber Components

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(J) Abstract

The protection offered by oxide films and adhering corrosion products during static tests (see Parts 1 and 2) was not reproducible under combustion conditions. The action of fast-moving gas and/or liquid droplets of metaphosphoric acid led to extremely rapid and high metal loss in the combustion system by erosion as well as corrosion.

This report describes the microscopical and surface characteristics of combustion components after exposure to the phosphorus-phosphorus sesquisulphide (PPS) combustion products. The examinations were carried out at various stages of the programme, principally to give some indication as to the cause of damage.

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